

## PATENT COOPERATION TREATY

PCT

## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents  
United States Patent and Trademark  
Office  
Box PCT  
Washington, D.C.20231  
ÉTATS-UNIS D'AMÉRIQUE

in its capacity as elected Office

<b>Date of mailing (day/month/year)</b> 10 February 2000 (10.02.00)	
<b>International application No.</b> PCT/AU99/00546	<b>Applicant's or agent's file reference</b> FP11220
<b>International filing date (day/month/year)</b> 07 July 1999 (07.07.99)	<b>Priority date (day/month/year)</b> 07 July 1998 (07.07.98)
<b>Applicant</b> RAJAKUMAR, Viruthiamparambath et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:

21 January 2000 (21.01.00)

☐ in a notice effecting later election filed with the International Bureau on:2. The election ☒ was☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

<b>The International Bureau of WIPO</b> 34, chemin des Colombettes 1211 Geneva 20, Switzerland	<b>Authorized officer</b>  Olivia RANAIVOJAONA
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

**PATENT COOPERATION TREATY**  
**PCT**  
**INTERNATIONAL PRELIMINARY EXAMINATION REPORT**  
(PCT Article 36 and Rule 70)

Applicant's or agent's file reference FP11220	<b>FOR FURTHER ACTION</b>	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416).
International application No. <b>PCT/AU99/00546</b>	International filing date ( <i>day/month/year</i> ) 7 July 1999	Priority Date ( <i>day/month/year</i> ) 7 July 1998
International Patent Classification (IPC) or national classification and IPC  Int. Cl. <sup>7</sup> C01B 7/03, C01G 49/06, C22B 34/12		
Applicant COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION et al		

1.	This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.																								
2.	This REPORT consists of a total of 3 sheets, including this cover sheet.  <input type="checkbox"/> This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).  These annexes consist of a total of     sheet(s).																								
3. This report contains indications relating to the following items: <table style="width: 100%; margin-top: 10px;"> <tr> <td style="width: 5%;">I</td> <td style="width: 5%; text-align: center;"><input checked="" type="checkbox"/></td> <td>Basis of the report</td> </tr> <tr> <td>II</td> <td style="text-align: center;"><input type="checkbox"/></td> <td>Priority</td> </tr> <tr> <td>III</td> <td style="text-align: center;"><input type="checkbox"/></td> <td>Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</td> </tr> <tr> <td>IV</td> <td style="text-align: center;"><input type="checkbox"/></td> <td>Lack of unity of invention</td> </tr> <tr> <td>V</td> <td style="text-align: center;"><input checked="" type="checkbox"/></td> <td>Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</td> </tr> <tr> <td>VI</td> <td style="text-align: center;"><input type="checkbox"/></td> <td>Certain documents cited</td> </tr> <tr> <td>VII</td> <td style="text-align: center;"><input type="checkbox"/></td> <td>Certain defects in the international application</td> </tr> <tr> <td>VIII</td> <td style="text-align: center;"><input type="checkbox"/></td> <td>Certain observations on the international application</td> </tr> </table>		I	<input checked="" type="checkbox"/>	Basis of the report	II	<input type="checkbox"/>	Priority	III	<input type="checkbox"/>	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability	IV	<input type="checkbox"/>	Lack of unity of invention	V	<input checked="" type="checkbox"/>	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement	VI	<input type="checkbox"/>	Certain documents cited	VII	<input type="checkbox"/>	Certain defects in the international application	VIII	<input type="checkbox"/>	Certain observations on the international application
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VI	<input type="checkbox"/>	Certain documents cited																							
VII	<input type="checkbox"/>	Certain defects in the international application																							
VIII	<input type="checkbox"/>	Certain observations on the international application																							

Date of submission of the demand 21 January 2000	Date of completion of the report 24 July 2000
Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized Officer  <b>M.R. OLLEY</b>  Telephone No. (02) 6283 2143

**I. Basis of the report****1. With regard to the elements of the international application:\***

- ☒ the international application as originally filed.
- ☐ the description,        pages , as originally filed,  
   pages , filed with the demand,  
   pages , received on    with the letter of
- ☐ the claims,        pages , as originally filed,  
   pages , as amended (together with any statement) under Article 19,  
   pages , filed with the demand,  
   pages , received on    with the letter of
- ☐ the drawings,        pages , as originally filed,  
   pages , filed with the demand,  
   pages , received on    with the letter of
- ☐ the sequence listing part of the description:  
   pages , as originally filed  
   pages , filed with the demand  
   pages , received on    with the letter of

**2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.**

These elements were available or furnished to this Authority in the following language which is:

- ☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

**3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, was on the basis of the sequence listing:**

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

**4. ☐ The amendments have resulted in the cancellation of:**

- ☐ the description,        pages
- ☐ the claims,        Nos.
- ☐ the drawings,        sheets/fig.

**5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).\*\***

\* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

\*\* Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement****1. Statement**

Novelty (N)	Claims 1-12	YES
	Claims	NO
Inventive step (IS)	Claims 1-12	YES
	Claims	NO
Industrial applicability (IA)	Claims 1-12	YES
	Claims	NO

**2. Citations and explanations (Rule 70.7)**

AU 30628/77

AU 46786/79

US 4994255

The above documents are prior art only and do not disclose all the features of the claimed invention. Therefore the claims are novel and have an inventive step.

09/720098

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Claims:

1. A process for recovering chlorine from chlorinator waste including the steps of:
  - 5 forming a fluidised bed of chlorinator waste in a fluidising gas containing oxygen and treating the chlorinator waste with oxygen under conditions which promote conversion of metal chlorides into metal oxides and discourage oxidation of carbon contained in the waste.
- 10 2. A process according to claim 1, wherein the conditions which promote conversion of metal chlorides into metal oxides and discourage oxidation of carbon contained in the waste are obtained by controlling the superficial velocity of the fluidising gas, the proportion of oxygen in  
15 the gas fed to the fluidised bed, oxygen to chlorinator waste feed ratio and temperature within the fluidised bed either separately or in combination.
3. A process according to claim 2, wherein the temperature of the fluidised bed is maintained in a range  
20 from 400 to 700°C.
4. A process according to claim 2 [or claim 3], wherein the superficial velocity of the gas is maintained in a range from 0.2 to 1 metre/second.
5. A process according to <sup>claim 2 - -</sup>any one of claims 2 to 4,  
25 wherein the stoichiometric ratio, R, is maintained in a range from 0.2 to 1.2.
6. A process according to <sup>claim 2 - -</sup>any one of claims 2 to 5, wherein the temperature of the fluidised bed is maintained in a range from 550 to 650°C.
- 30 7. Apparatus for recovering chlorine from chlorinator waste, which apparatus includes a fluidised bed reactor, means for introducing chlorinator waste into the fluidised bed reactor, means for introducing a fluidising gas containing oxygen into the fluidised bed reactor and  
35 means for controlling oxygen to chlorinator waste molar feed ratio, superficial velocity of fluidising gas, proportion of oxygen in the fluidising gas and temperature

## ABSTRACT

The specification discloses a process for recovering chlorine from a chlorinator waste. The process involves treating the chlorinator waste with oxygen in a fluidised bed under conditions which promote the conversion of metal chlorides to metal oxides and  
10 discourage the oxidation of carbon contained in the waste. Suitable conditions include a bed temperature in a range from 400 to 700°C, a superficial velocity in a range from 0.2 to 1 metre/second and stoichiometric ratio, R, in a range from 0.2 to 1.2.

## PCT REQUEST

FP11220

Original (for SUBMISSION) - printed on 07.07.1999 09:44:24 AM

0	For receiving Office use only	
0-1	International Application No.	
0-2	International Filing Date	
0-3	Name of receiving Office and *PCT International Application*	
0-4	Form - PCT/RO/101 PCT Request Prepared using	PCT-EASY Version 2.84 (updated 01.06.1999)
0-5	Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
0-6	Receiving Office (specified by the applicant)	Australian Patent Office (RO/AU)
0-7	Applicant's or agent's file reference	FP11220
I	Title of invention	PROCESS FOR CHLORINE RECOVERY
II	Applicant	
II-1	This person is:	applicant only
II-2	Applicant for	all designated States except US
II-4	Name	COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION
II-5	Address:	LIMESTONE AVENUE CAMPBELL, Australian Capital Territory 2612 Australia
II-6	State of nationality	AU
II-7	State of residence	AU
III-1	Applicant and/or inventor	
III-1-1	This person is:	applicant and inventor
III-1-2	Applicant for	US only
III-1-4	Name (LAST, First)	RAJAKUMAR, Viruthiamparambath
III-1-5	Address:	c/o CSIRO Division of Minerals PO BOX 312 Rosebank MDC CLAYTON, Victoria 3169 Australia
III-1-6	State of nationality	AU
III-1-7	State of residence	AU

## PCT REQUEST

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FP11220

Original (for SUBMISSION) - printed on 07.07.1999 09:44:24 AM

III-2	<b>Applicant and/or inventor</b>	
III-2-1	This person is:	applicant and inventor
III-2-2	Applicant for	US only
III-2-4	Name (LAST, First)	BOWYER O'CONNELL HARDING, Damien
III-2-5	Address:	c/o CSIRO Division of Minerals PO Box 312 Rosebank MDC CLAYTON, Victoria 3169 Australia
III-2-6	State of nationality	AU
III-2-7	State of residence	AU
IV-1	<b>Agent or common representative; or address for correspondence</b> The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:	agent
IV-1-1	Name	GRIFFITH HACK
IV-1-2	Address:	509 ST KILDA ROAD MELBOURNE, Victoria 3004 Australia
IV-1-3	Telephone No.	03 9243 8300
IV-1-4	Facsimile No.	03 9243 8333
IV-1-5	e-mail	ghmelb@griffithhack.com.au
V	<b>Designation of States</b>	
V-1	Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AP: GH GM KE LS MW SD SL SZ UG ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT EP: AT BE CH&LI CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE and any other State which is a Contracting State of the European Patent Convention and of the PCT OA: BF BJ CF CG CI CM GA GN GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT
V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AE AL AM AT AU AZ BA BB BG BR BY CA CH&LI CN CU CZ DE DK EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT UA UG US UZ VN YU ZA ZW



## PCT REQUEST

FP11220

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V-5	<b>Precautionary Designation Statement</b> In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.		
V-6	<b>Exclusion(s) from precautionary designations</b>	NONE	
VI-1	<b>Priority claim of earlier national application</b>		
VI-1-1	Filing date	07 July 1998 (07.07.1998)	
VI-1-2	Number	PP4550	
VI-1-3	Country	AU	
VI-2	<b>Priority document request</b> The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) identified above as item(s):	VI-1	
VII-1	<b>International Searching Authority Chosen</b>	Australian Patent Office (ISA/AU)	
VIII	<b>Check list</b>	number of sheets	electronic file(s) attached
VIII-1	Request	4	-
VIII-2	Description	21	-
VIII-3	Claims	2	-
VIII-4	Abstract	1	abstract_fp11220.txt
VIII-5	Drawings	6	-
VIII-7	TOTAL	34	
VIII-8	<b>Accompanying items</b>	paper document(s) attached	electronic file(s) attached
VIII-16	Fee calculation sheet	✓	-
VIII-16	PCT-EASY diskette	-	diskette
VIII-18	<b>Figure of the drawings which should accompany the abstract</b>	5	
VIII-19	<b>Language of filing of the international application</b>	English	
IX-1	<b>Signature of applicant or agent</b>		
IX-1-1	Name	GRIFFITH HACK	
IX-1-2	Name of signatory	John Graeme Blair	

## PCT REQUEST

FP11220

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## FOR RECEIVING OFFICE USE ONLY

10-1	Date of actual receipt of the purported international application	
10-2	Drawings:	
10-2-1	Received	
10-2-2	Not received	
10-3	Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application	
10-4	Date of timely receipt of the required corrections under PCT Article 11(2)	
10-5	International Searching Authority	ISA/AU
10-6	Transmittal of search copy delayed until search fee is paid	

## FOR INTERNATIONAL BUREAU USE ONLY

11-1	Date of receipt of the record copy by the International Bureau	
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# PCT

## POWER OF ATTORNEY

(for an international application filed under the Patent Cooperation Treaty)

(PCT Rule 90.4)

The undersigned applicant(s) (Names should be indicated as they appear in the request):

COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION  
LIMESTONE AVENUE  
CAMPBELL ACT 2612  
AUSTRALIA

hereby appoints (appoint) the following person as:

☒ agent

☐ common representative

Name and address

(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

BLAIR, John Graeme  
509 ST KILDA ROAD  
MELBOURNE VIC 3004  
AUSTRALIA

to represent the undersigned before

☒ all the competent International Authorities

☐ the International Searching Authority only

☐ the International Preliminary Examining Authority only

in connection with the international application identified below:

Title of the invention: PROCESS FOR CHLORINE RECOVERY

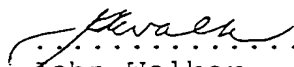
Applicant's or agent's file reference: FP11220

International application number (if already available):

Filed with the following Office IP AUSTRALIA as receiving Office  
and to make or receive payments on behalf of the undersigned.

Signature of the applicant(s) (where there are several applicants, each of them must sign; next to each signature, indicate the name of the person signing and the capacity in which the person signs, if such capacity is not obvious from reading the request or this power):

COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION

  
.....  
John Walker  
Authorised Officer

Date: 13-7-99

# PCT

## POWER OF ATTORNEY

(for an international application filed under the Patent Cooperation Treaty)

(PCT Rule 90.4)

The undersigned applicant(s) (Names should be indicated as they appear in the request):

RAJAKUMAR, Viruthiamparambath  
c/o CSIRO Division of Minerals  
PO Box 312 Rosebank MDC  
CLAYTON, Victoria 3169  
AUSTRALIA

hereby appoints (appoint) the following person as:

☒ agent

☐ common representative

Name and address

(Family name followed by given name: for a legal entity, full official designation. The address must include postal code and name of country.)

BLAIR, John Graeme  
GRIFFITH HACK of  
3<sup>rd</sup> Floor, 509 St Kilda Road, Melbourne Victoria 3004, Australia

to represent the undersigned before



all the competent International Authorities



the International Searching Authority only



the International Preliminary Examining Authority only

in connection with the international application identified below:

Title of the invention: PROCESS FOR CHLORINE RECOVERY

Applicant's or agent's file reference: FP11220

International application number (if already available):

filed with the following Office Australian Industrial Property Organisation as receiving Office and to make or receive payments on behalf of the undersigned.

Signature of the applicant(s) (where there are several applicants, each of them must sign; next to each signature, indicate the name of the person signing and the capacity in which the person signs, if such capacity is not obvious from reading the request or this power):

Signature:

Viruthiamparambath Rajakumar

Date:

15/9/99

Name: Viruthiamparambath RAJAKUMAR

Position:

The demand must be filed directly with the competent International Preliminary Examining Authority or, if two or more Authorities are competent, with the one chosen by the applicant. The full name or two-letter code of that Authority may be indicated by the applicant on the line below:

IPEA/ \_\_\_\_\_

# PCT

## CHAPTER II

### DEMAND

under Article 31 of the Patent Cooperation Treaty:  
The undersigned requests that the international application specified below be the subject of international preliminary examination according to the Patent Cooperation Treaty and hereby elects all eligible States (except where otherwise indicated).

For International Preliminary Examining Authority use only	
Identification of IPEA	Date of receipt of DEMAND
<b>Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION</b>	
Applicant's or agent's file reference FP11220	
International application No. PCT/AU99/00546	International filing date (day/month/year) 7/7/99
(Earliest) Priority date (day/month/year) 7/7/98	
Title of invention PROCESS FOR CHLORINE RECOVERY	
<b>Box No. II APPLICANT(S)</b>	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)	
COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION Limestone Avenue CAMPBELL ACT 2612 AUSTRALIA	
Telephone No.:	
Facsimile No.:	
Teleprinter No.:	
State (that is, country) of nationality: AUSTRALIA	State (that is, country) of residence: AUSTRALIA
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)	
RAJAKUMAR, Viruthiamparambath c/o CSIRO DIVISION OF MINERALS PO Box 312, Rosebank MDC CLAYTON VIC 3169 AUSTRALIA	
State (that is, country) of nationality: AUSTRALIA	State (that is, country) of residence: AUSTRALIA
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)	
BOWYER O'CONNELL HARDING, Damien c/o CSIRO DIVISION OF MINERALS PO Box 312, Rosebank MDC CLAYTON VIC 3169 AUSTRALIA	
State (that is, country) of nationality: AUSTRALIA	State (that is, country) of residence: AUSTRALIA
<input type="checkbox"/> Further applicants are indicated on a continuation sheet.	

**Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE**The following person is ☒ agent ☐ common representativeand ☒ has been appointed earlier and represents the applicant(s) also for international preliminary examination.☐ is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked.☐ is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.Name and address: *(Family name followed by given name; for a legal entity, full official designation.  
The address must include postal code and name of country.)*BLAIR, John Graeme  
GRIFFITH HACK  
509 St Kilda Road  
MELBOURNE VIC 3004  
AUSTRALIA

Telephone No.:

+61 3 9243 8300

Facsimile No.:

+61 3 9243 8333/4

Teleprinter No.:

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.**Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION****Statement concerning amendments:\***

1. The applicant wishes the international preliminary examination to start on the basis of:

☒ the international application as originally filedthe description ☐ as originally filed☐ as amended under Article 34the claims ☐ as originally filed☐ as amended under Article 19 (together with any accompanying statement)☐ as amended under Article 34the drawings ☐ as originally filed☐ as amended under Article 342. ☐ The applicant wishes any amendment to the claims under Article 19 to be considered as reversed.3. ☐ The applicant wishes the start of the international preliminary examination to be postponed until the expiration of 20 months from the priority date unless the International Preliminary Examining Authority receives a copy of any amendments made under Article 19 or a notice from the applicant that he does not wish to make such amendments (Rule 69.1(d)). *(This check-box may be marked only where the time limit under Article 19 has not yet expired.)*

\* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purposes of international preliminary examination: English☒ which is the language in which the international application was filed.☐ which is the language of a translation furnished for the purposes of international search.☐ which is the language of publication of the international application.☐ which is the language of the translation (to be) furnished for the purposes of international preliminary examination.**Box No. V ELECTION OF STATES**The applicant hereby elects all eligible States *(that is, all States which have been designated and which are bound by Chapter II of the PCT)*

excluding the following States which the applicant wishes not to elect:

## Box No. VI CHECK LIST

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination:

- |  |   |        |
|--|---|--------|
| 1. translation of international application                              | : | sheets |
| 2. amendments under Article 34   | : | sheets |
| 3. copy (or, where required, translation) of amendments under Article 19 | : | sheets |
| 4. copy (or, where required, translation) of statement under Article 19  | : | sheets |
| 5. letter  | : | sheets |
| 6. other (specify)   | : | sheets |

For International Preliminary Examining Authority use only

received not received

<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>

The demand is also accompanied by the item(s) marked below:

- |  |   |
|--|---|
| 1. <input type="checkbox"/> fee calculation sheet  | 4. <input type="checkbox"/> statement explaining lack of signature                                  |
| 2. <input type="checkbox"/> separate signed power of attorney                            | 5. <input type="checkbox"/> nucleotide and or amino acid sequence listing in computer readable form |
| 3. <input type="checkbox"/> copy of general power of attorney; reference number, if any: | 6. <input type="checkbox"/> other (specify):  |

## Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand).

COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION



John Graeme Blair  
For and on behalf of the Applicants

20/01/2000  
Date

For International Preliminary Examining Authority use only

1. Date of actual receipt of DEMAND:

2. Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):

3. ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply.

☐ The applicant has been informed accordingly.

4. ☐ The date of receipt of the demand is WITHIN the period of 19 months from the priority date as extended by virtue of Rule 80.5.

5. ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.

For International Bureau use only

Demand received from IPEA on:

# PATENT COOPERATION TREATY

From the:  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

<p>To: GRIFFITH HACK GPO Box 1285K MELBOURNE VIC 3001 [g3]</p> <p style="font-size: 2em; opacity: 0.5; transform: rotate(-15deg); position: absolute; left: 100px; top: 100px;">DIARIED</p> <p style="position: absolute; left: 140px; top: 170px;">ID.....146249.....</p>	<p>GRIFFITH HACK</p> <p>02 MAR 2000</p> <p><i>lets</i></p> <p>JGB</p>
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PCT

WRITTEN OPINION

(PCT Rule 66)

Date of mailing (day/month/year)	02 March 2000
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Applicant's or agent's file reference JGB: LM: FP11220	REPLY DUE within <b>TWO MONTHS</b> from the above date of mailing
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International application No. <b>PCT/AU 99/00546</b>	International filing date (day/month/year) <b>07 July 1999</b>	Priority Date (day/month/year) <b>07 July 1998</b>
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International Patent Classification (IPC) or both national classification and IPC <b>Int. Cl.<sup>7</sup> C01B 7/03, C01G 49/06, C22B 34/12</b>
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Applicant
1. COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION
2. BOWYER O'CONNELL HARDING, Damien
3. RAJAKUMAR, Viruthiamparambath

1.	This written opinion is the <b>FIRST</b> (first, etc) drawn by this International Preliminary Examining Authority.
2.	This opinion contains indications relating to the following items:
I	<input checked="" type="checkbox"/> Basis of the opinion
II	<input type="checkbox"/> Priority
III	<input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
IV	<input type="checkbox"/> Lack of unity of invention
V	<input checked="" type="checkbox"/> Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
VI	<input type="checkbox"/> Certain documents cited
VII	<input type="checkbox"/> Certain defects in the international application
VIII	<input type="checkbox"/> Certain observations on the international application
3.	The applicant is hereby invited to reply to this opinion.
When?	See the time limit indicated above. The applicant may, before the expiration of that time limit, request this Authority to grant an extension, see Rule 66.2(d).
How?	By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.
Also	For an additional opportunity to submit amendments, see Rule 66.4. For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4bis. For an informal communication with the examiner, see Rule 66.6.
If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.	
4.	The final date by which the international preliminary examination report must be established according to Rule 69.2 is: <b>07 November 2000</b>

<p>Name and mailing address of the IPEA/AU</p> <p>AUSTRALIAN PATENT OFFICE PO BOX 200 WODEN ACT 2606 AUSTRALIA E-mail address: <a href="mailto:pct@ipaaustralia.gov.au">pct@ipaaustralia.gov.au</a> Facsimile No. (02) 6285 3929</p>	<p>Authorized Officer</p> <p style="text-align: center;"><i>M. Bremer</i></p> <p>for <b>NEROLIE A. BUSH</b> Telephone No. (02) 6283 2142</p>
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**I. Basis of the opinion**

**1. With regard to the elements of the international application:\***

- ☒ the international application as originally filed.
- ☐ the description,        pages    , as originally filed,  
    pages    , filed with the demand,  
    pages    , filed with the letter of    .
- ☐ the claims,        pages    , as originally filed,  
    pages    , as amended under Article 19,  
    pages    , filed with the demand,  
    pages    , filed with the letter of    .
- ☐ the drawings,        pages    , as originally filed,  
    pages    , filed with the demand,  
    pages    , filed with the letter of    .
- ☐ the sequence listing part of the description:  
    pages    , as originally filed  
    pages    , filed with the demand  
    pages    , filed with the letter of    .

**2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.**

These elements were available or furnished to this Authority in the following language    which is:

- ☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

**3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the written opinion was drawn on the basis of the sequence listing:**

- ☐ contained in the international application in printed form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

**4. The amendments have resulted in the cancellation of:**

- ☐ the description,        pages
- ☐ the claims,        Nos.
- ☐ the drawings,        sheets/fig

**5. ☐ This opinion has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

*\* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed"*

**V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement****1. Statement**

Novelty (N)	Claims	YES
	Claims 1-12	NO
Inventive step (IS)	Claims	YES
	Claims 1-12	NO
Industrial applicability (IA)	Claims 1-12	YES
	Claims	NO

**2. Citations and explanations**NOVELTY (N) Claims 1-12:

- AU 30628/77 A
- AU 46786/79 A PAGE 4, LINE 45 TO LINE 68
- US 4994255 A Column 6 line 15 to Column 7 line 15, claim 1

Each of these citations explicitly discloses all of the features of the claims 1-12.

For example AU 30628/77 A

- Fluidising bed of chlorinator waste, page 5 line 19-23
- Treat chlorinator waste with oxygen, page 6, lines 1-4
- Conversion of metal chlorides into metal oxides, no oxidation of carbon, page 6, line 4.
- Reaction conditions that discourage oxidation of carbon controlled by proportion of oxygen, oxygen to chlorinator ratio, temperature, page 6, line 21 to 23, page 6 line 3.

INVENTIVE STEP (IS) Claims 1-12:

Claims 1 to 12 as above.

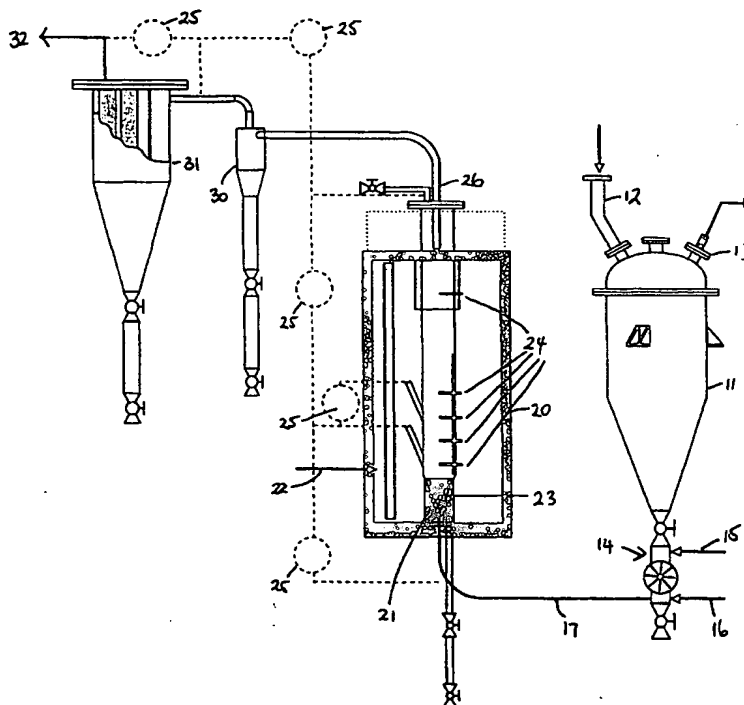


## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>6</sup> :</b> <b>C01B 7/03, C01G 49/06, C22B 34/12</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 00/02813</b>
		<b>(43) International Publication Date:</b> 20 January 2000 (20.01.00)
<p><b>(21) International Application Number:</b> PCT/AU99/00546</p> <p><b>(22) International Filing Date:</b> 7 July 1999 (07.07.99)</p> <p><b>(30) Priority Data:</b>          PP 4550                      7 July 1998 (07.07.98)                      AU</p> <p><b>(71) Applicant (for all designated States except US):</b> COMMON-WEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION [AU/AU]; Limestone Avenue, Campbell, ACT 2612 (AU).</p> <p><b>(72) Inventors; and</b>  <b>(75) Inventors/Applicants (for US only):</b> RAJAKUMAR, Viruthi-amparambath [AU/AU]; (AU). BOWYER O'CONNELL HARDING, Damien [AU/AU]; CSIRO Division of Minerals, P.O. Box 312 Rosebank MDC, Clayton, VIC 3169 (AU).</p> <p><b>(74) Agent:</b> GRIFFITH HACK; 509 St. Kilda Road, Melbourne, VIC 3004 (AU).</p>		<p><b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b>  <i>With international search report.</i></p>

**(54) Title:** PROCESS FOR CHLORINE RECOVERY**(57) Abstract**

The specification discloses a process for recovering chlorine from a chlorinator waste. The process involves treating the chlorinator waste with oxygen in a fluidised bed under conditions which promote the conversion of metal chlorides to metal oxides and discourage the oxidation of carbon contained in the waste. Suitable conditions include a bed temperature in a range from 400 to 700 °C, a superficial velocity in a range from 0.2 to 1 metre/second and a stoichiometric ratio, R, in a range from 0.2 to 1.2.



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PROCESS FOR CHLORINE RECOVERY

The present invention is concerned with a process for the recovery of chlorine from metal chlorides. More specifically, the invention relates to a process for dechlorinating iron chloride mixed with several other metal chlorides to produce chlorine gas. The process has the ability to recover chlorine from these chlorides in the presence of carbon and other materials such as oxides which are present in typical wastes generated during the chlorination of synthetic rutile.

Titanium tetrachloride is conventionally produced from either ilmenite or rutile (including synthetic rutile) in a fluidised bed at around 1000°C. Chlorine, a carbon rich material such as petroleum coke and a titanium-bearing material are fed into the fluidised bed chlorinator and titanium tetrachloride vapour leaves the system in the gas phase. It is subsequently condensed, purified and used in the production of either pigment or titanium metal. Chlorine is recycled to the chlorinator.

Impurities such as Mg, Mn, Al in the feed are chlorinated to varying degrees. Iron in the feed chlorinates readily and is sufficiently volatile to leave the reactor with the effluent gas. The chlorides of these metals are condensed from the gas phase at temperatures around 200°C, while titanium tetrachloride, which has a lower boiling point, remains as a vapour. Non titanium metal chlorides are therefore amenable to separation by differential condensation. They are recovered as solids in the chlorinator off gas cooling system.

The condensed stream from the chlorinator typically contains chlorides such as  $\text{FeCl}_2$ ,  $\text{MnCl}_2$ ,  $\text{MgCl}_2$  and  $\text{AlOCl}$ , as well as large quantities of coke and synthetic rutile which are blown over from the chlorinator. This material is referred to as the chlorinator waste.

The chlorinator waste is subsequently disposed of by whatever means are most acceptable from an environmental

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point of view. Techniques include direct dumping in mineshafts, blending into concrete for low-strength marine applications and wet chemical treatment to produce iron oxide and HCl (or NaCl with usage of caustic soda). In some pigment plants the waste is currently disposed of by mixing it with CaO and water which react with the chlorides to form oxides and  $\text{CaCl}_2$ . The oxides and other solids are thickened, de-watered and returned to the mine. The  $\text{CaCl}_2$  liquor is discharged into the ocean.

These forms of disposal mean that valuable chlorine present as chlorides in the chlorinator waste is not recovered. These options are also undesirable because they are either environmentally sensitive or costly in terms of consumables and generally call for minimisation of iron chloride production. For this reason, rutile is the feedstock of choice for chlorination. It contains less iron than ilmenite. In commercial processes, therefore, ilmenite is first converted to synthetic rutile by removing substantially all of the iron by appropriate pre-treatment processes and the synthetic rutile is subsequently used in the chlorination process.

An alternative to simply disposing of the chlorinator waste is to react the waste directly with oxygen to form oxides and recover the chlorine as shown schematically in Figure 1. The oxides generated could be useful as landfill or smelter feed, or returned to the mine and the chlorine recycled to the chlorinator. Such a dechlorination process has the potential to reduce the cost of pigment production by reducing the quantity of fresh chlorine required, and by significantly reducing the consumption of water.

The chlorination industry has a long-standing need for a process which can convert iron chloride into chlorine and iron oxide. In the production of titanium tetrachloride the chlorine could be directly recycled to the chlorinator, thereby decreasing the need for chlorine-make-up. For such a process to be effective, the iron oxide

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produced would need to be sufficiently free of chlorine to allow disposal as landfill or smelter feed with little or no further treatment. The availability of such a process would create the potential for processes such as:

5           Direct chlorination of ilmenite in the case of "clean" (low Ca, Mg, Mn, Al) deposits. This simultaneously obviates the need for synthetic rutile production and offers a solution to the iron chloride disposal problem.

10           Ilmenite conversion to synthetic rutile by partial chlorination with subsequent alkali removal in the case of dirty (high Ca, Mg and/or Mn) deposits. This would present an alternative to more conventional methods of impurity removal by reduction and leaching and would lead to reduced waste disposal requirements.

15           A general waste disposal route for undesired iron chloride and other chlorides which yields chlorine for direct recycle to chlorination systems. This includes the use of skid mounted plants to process accumulated iron chloride waste at existing chlorinator sites, particularly  
20           in Europe and USA.

#### Prior art

          The proposed technologies for chlorine recovery, as disclosed in the literature, are not sufficiently  
25           selective and/or have unattractive scale-up features.

          The Du Pont recirculating fluidised bed approach (US patents 3793444, 4144316 and 4174381) claims greater than 95% chlorine removal, leaving a nominal 3-5% leachable chloride in the solid phase and rendering it unsuitable for  
30           direct disposal.

          The Mitsubishi vapour-phase approach (S Fukushima and Y. Sugawara, Light metals, AIME 1974), claims 90% chlorine removal and in addition, has perceived scale-up limitations.

35           The Mineral Process approach (US patent 4140746) comprises partial dechlorination of ferric chloride to ferrous chloride in the presence of a reducing agent such

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as sulfur or chlorine polysulfides to produce a chloride compound in the first step. In the second step the ferrous chloride is oxidised to ferric chloride and ferric oxide and the ferric chloride is recycled to the first step. The  
5 chlorine values were recovered as compounds containing chlorine but not as chlorine gas.

SCM Chemical's US patent 4624843 (inventor M Robinson, 25 Nov 1986) refers to control of the proportions of the iron chloride and carbon in the blown over material  
10 from the chlorinator by controlling the iron oxide concentration in the feed to the chlorinator. The iron oxide is claimed to be introduced through the addition of ilmenite and/or rutile slag. The chlorinator is therefore being used as a selective chlorinator or beneficiator to  
15 upgrade other Ti bearing materials. The blown over carbon is controlled between 7.5 and 20% carbon based on the carbon plus iron chloride only, the blow over of rutile is not specifically mentioned. It is claimed that the iron chloride is formed as ferric chloride under the conditions  
20 used in the selective chlorinator.

The dechlorinator claimed in US patent 4624843 is based on the introduction of more than one oxygen stream into the reactor. A first stream introduced at the base of the fluidised bed is intended to react with the carbon  
25 content of the feed and maintain the bed temperature at between 500 and 1050°C, "preferably at least at 600°C". This step seems to be intended to vaporise the ferric chloride although the patent is not very clear on this aspect. A second or more oxygen streams, which appear to  
30 need preheating, are introduced above the bed to react with the ferric chloride vapour.

With the dechlorination reaction occurring in the gas phase above the bed between ferric chloride vapour and oxygen, the conversions are unlikely to be complete because  
35 of residence time constraints. This implies that iron chloride recycle levels will be significant. Moreover, the iron oxide will be produced as fine dust which increase the



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probability of accretion formation at the bed exit.

We have discovered that waste obtained from typical chlorinators a) the iron is present as ferrous chloride, b) the conversion of ferrous to ferric chloride is reasonably rapid during dechlorination and c) the conversion of chloride to oxide is helped by using a fluidised bed of particles. The need for the type of pseudo multi-stage operation claimed in US patent 4624843 makes reactor operation and control more difficult with no perceived gain.

SCM Chemicals' US patent 4994255 (inventor C K Hahn, 19 February 1991) is a process patent which claims several hypothetical process schemes in which a major feature is the claim to apparent complete or near complete separation of carbon from the rest of the blown over material. It is claimed that "the carbon and ore solids can be most efficiently removed by using a ceramic filter bag in a baghouse. The temperature can be as high as 800°C." However, the patent goes on to state that the "carbon content in the condensed FeCl<sub>2</sub> under these conditions is below about 12% and usually, the removal of carbon and ore is almost complete". The patent also refers to US patent 4094954 and states that "the carbon, together with any ore in the offgas stream can be separated by conventional means such as using a hot cyclone separator, electrostatic separator or a knock-out pot. Their claim implies, erroneously, that at 800°C the ferrous chloride is in vapour form.

The conceptual flowsheets in figures 1,2,4 and 5 in US patent 4994255 are not substantiated by any results. It is clear from the descriptions and diagrams that the patent assumes that substantially pure FeCl<sub>2</sub> is treated in the FeCl<sub>2</sub> oxidiser. The FeCl<sub>2</sub> oxidiser appears to be based on an inert material which is partly removed with the iron oxide coating. Because their patent is based on pure or substantially pure iron chloride, an inert bed is necessary to avoid potential problems due to molten chlorides.

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US patent 4994255 does not reveal the optimum operating conditions such as temperature and oxygen/waste feed ratio for maximum chlorine recovery at economic rates of chlorine production other than to say that the  
5 temperature should be below the melting point of  $\text{FeCl}_2$  ( $674^\circ\text{C}$ ).

We have discovered that in a real chlorinator waste the conversion rates of the chlorides can be quite rapid despite the presence of liquid phases which melt at  
10 much lower temperatures when other metal chlorides such as those of Mn and Mg are present along with iron chloride. Our invention is not limited to pure iron chloride.

US patent 4144316 for ferric chloride conversion claims the need to add a catalyst (sodium chloride) to  
15 achieve satisfactory conversion in a fluidised bed of ferric chloride particles. US patent 3944647, restricted to the treatment of ferric chloride, also claims the use of sodium chloride to produce a liquid sodium ferric chloride salt complex.

20 Various publications have dealt with the problems of obtaining high conversions with ferric chloride. A paper by Olsen (Olsen R S, in Recycle and Secondary recovery of metals, 1985, Taylor P R et al editors, pp771-783) gives results of a preliminary study of the treatment of ferrous  
25 chloride in a fluidised bed but the reported conversions were low. We have discovered that in a real chlorinator waste, the conversion rate and final conversion of the chlorides in the waste are high without the need for any catalyst addition.

30 US patent 3642441 (Falconbridge Nickel Mines) appears to be based on a complex process with a multiplicity of streams and relies on the endothermic reaction of "explosive" mixtures of metal chlorides with water and combustion of the mixtures. Significant  
35 quantities of hydrogen bearing gases will result and even if the process were capable of being scaled up satisfactorily, it is likely to be more suited to  $\text{HCl}$

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rather than chlorine production.

US patent 3325252, and related US patent 3050365, are based on a two-zone furnace using molten iron chloride feed. Based on the quoted example, liquid ferric chloride is fed under pressure through a spray head to the inner tube of a burner with oxygen supply through an outer tube. It is claimed that the reaction is completed in a second, lower zone, into which alumina particles are fed. This zone was maintained at about 450°C but the rate of chlorine recovery would have been low under these conditions. The process seems to be restricted to iron chloride levels less than 10% in the feed. Higher ferrous chloride levels are unlikely to give satisfactory chlorine recoveries because of short residence times and the absence of inert substrate particles in the upper zone and the slow kinetics in the lower zone. Accretion formation is also seen as a major problem. US patent 3092456 attempts to address this issue but a practical solution is not simple.

The vapour phase contacting method claimed by Mitsubishi (US patent 4073874 to Fukushima et al) is based on treating ferric chloride. The ferric chloride vapour and several streams of oxygen, injected through nozzles, react with each other in an enclosure. It is claimed that in initial work there were significant operational problems caused by the formation of iron oxide accretions at the point where the chloride vapour stream impinged on the oxygen stream. It appears that the problem (Fukushima S and Sugawara Y in Light Metals Technology II, 1974, pp443-466) with accretions was not completely eliminated.

In summary, the application of fluidised bed or other (mainly vapour phase contacting with oxygen) technologies to the recovery of chlorine from iron chloride has been the subject of a few patents and literature but these claimed processes have severe limitations that have apparently hindered successful commercial implementation.

Processes based on direct gas phase contacting between iron chloride vapour and oxygen in the absence of a

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bed of inert material have significant problems due to accretion formation resulting from the production of fine dust of iron oxide.

Beneficiation/partial chlorination in which iron units are added through ilmenite or rutile based slag to control the carbon/chloride ratio in the waste is impractical because control of the waste composition to optimise the dechlorination unit cannot be achieved as indicated in US patent 4624843.

Pre-oxidation of the waste to 500-800°C (US patent 4060584) to apparently convert the ferrous chloride to ferric chloride vapour and iron oxide, the ferric chloride vapour being separated from the iron oxide using high temperature separation and subsequently dechlorinating the iron chloride vapour in a multi stage process will not recover the significant amount of chlorine associated with the other metals which will stay with the solids in the pre-oxidiser.

It is also important to note that the patents ignore the fact that the chlorine associated with iron is only a proportion of the total chlorine available for recovery and the chlorine associated with the other elements such as Mn, Mg and Al and other elements need to be recovered.

We note that carbon and ore blow through occurs routinely in synthetic rutile chlorination plants and recognise that it is difficult to eliminate such blow through and/or implement cost-effective technology for separating the carbon and ore from the chlorides at the exit of the chlorinator. We have also discovered that the major amount of chloride collected in the blown over material is coated on the larger carbon and rutile particles with a minor amount as free chlorides. It is therefore important to recover the chlorine from the chloride coated on the particles and it is not sufficient to remove the carbon and rutile particles by filtration or other means as has been proposed in some of the prior art

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discussed above.

According to a first aspect of the present invention there is provided a process for recovering chlorine from chlorinator waste including the steps of forming a fluidised bed of chlorinator waste in a fluidising gas containing oxygen and treating the chlorinator waste with oxygen under conditions which promote conversion of metal chlorides into metal oxides and discourage oxidation of carbon contained in the waste.

Preferably the process further includes the steps of separately recovering metal oxides and chlorine gas. Typically the chlorine gas is recycled into the chlorinator in a process for the production of titanium tetrachloride from ilmenite or rutile, including synthetic rutile.

Typically the chlorinator waste contains chlorides such as  $\text{FeCl}_2$ ,  $\text{MnCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{AlOCl}$ , as well as large quantities of coke and feed material such as synthetic rutile which are blown over from the chlorinator in a process for production of titanium tetrachloride. Accordingly, in a preferred embodiment of the invention the dechlorination of the chloride is achieved by controlling superficial velocity of the fluidising gas in the fluidised bed, the proportion of oxygen in the gas fed to the fluidised bed, the oxygen to chlorinator waste feed ratio and the temperature within the fluidised bed, either separately or in combination, so as to maximize the recovery of chlorine gas and/or minimize the conversion of carbon. Under these conditions the dechlorination process strips chloride absorbed to particulate matter in the feed, such as carbon and rutile particles, and from separate chloride particles in the feed.

Preferably the temperature in the fluidised bed is in the range of 400 to 700°C, the superficial velocity of the gas is in the range of 0.2 to 1.0 m/s and the oxygen to chlorinator waste stoichiometric ratio,  $R$ , is in the range 0.2 to 1.2. The stoichiometric ratio,  $R$ , is the ratio of oxygen supplied to the stoichiometric oxygen

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required to completely convert the chlorides and carbon to obtain chlorine and carbon dioxide respectively. Typically the temperature in the fluidised bed reactor, the superficial velocity of the gas and the oxygen to waste molar ratio can be selected depending on the composition and morphological characteristics of the chlorides and other particles, including the particle size of the particles, in the chlorinator waste.

According to a second aspect of the present invention there is provided an apparatus for recovering chlorine from chlorinator waste, which apparatus includes fluidised bed reactor, means for introducing chlorinator waste into the fluidised bed reactor, means for introducing a fluidising gas containing oxygen into the fluidised bed reactor and means for controlling oxygen to chlorinator waste molar feed ratio, superficial velocity of fluidising gas and temperature within the fluidised bed reactor.

The fluidised bed reactor may have a conical bottom entry region for the chlorinator waste feed material and fluidising gas. Alternatively, the fluidising gas may be fed into the fluidised bed reactor through a distributor plate. The reactor is appropriately cooled or heated to allow the extraction or addition of heat to the process depending on the energy requirement of the process, which can be calculated based on the waste composition and the operating parameters.

According to a third aspect of the present invention there is provided a system for recovering chlorine from chlorinator waste including the apparatus described above, and further including means for collecting particulate matter which leaves the fluidised bed reactor and means for quenching any unreacted chlorinated compounds, particulates and the chlorine gas stream which leave the fluidised bed reactor.

Typically the means for collecting particulate matter is a cyclone and the means for quenching unreacted chlorinated compounds is a quencher downstream of the

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cyclone. In this arrangement solid materials are collected in the cyclone and the resultant gas stream passed through the quencher. The quencher is arranged so that condensable vapors are removed by quenching and the cooled chlorine gas stream that results is conveyed to a baghouse or other appropriate separation device to remove any residual fine dust particles so as to obtain a clean chlorine gas stream. The chlorine gas stream can be directed to a chlorinator in a titanium tetrachloride production process.

Preferably the cyclone is maintained at a temperature above the condensation temperature of any unreacted chlorinated compounds so that an effective separation is achieved between the solids and the unreacted chlorinated compounds. Typically the conditions in the cyclone, including the residence time of the particles and the temperature in the cyclone are chosen so as to avoid oxidation of carbon in the cyclone.

According to a fourth aspect of the present invention there is provided a process for the dechlorination of metal chlorides in a mixture including at least one metal chloride and also including carbon, or carbon-containing materials, and other materials such as, but not limited to, metal oxides, the process including forming a fluidised bed of the mixture in a fluidising gas containing oxygen and converting the mixture under conditions which promote conversion of metal chlorides to metal oxides and discourage oxidation of carbon.

Typically the mixture contains ferrous and/or ferric chloride and chlorides such as those of manganese, magnesium, calcium and aluminium. The mixture may also include metal chlorides such as those of V, Cr, Mb, Zr, Na, Ba, Ce, Sr, Si, Be or Cu.

Preferred embodiments of the present invention will be described, by way of example only, with reference to the accompanying figures, in which:

Fig 1 is a flow chart illustrating the general process for preparation of titanium dioxide pigment from

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synthetic rutile, including treatment of the chlorinator waste.

Fig 2 shows a back scatter electron image of (a) a synthetic rutile particle and (b) a carbon particle typical of those present in a sample of blown over material from an operating chlorinator in the process schematically illustrated in Fig 1.

Fig 3 is a graph of fraction of chlorine present as certain chosen species verses temperature for a thermodynamic simulation of the effect of temperature on the distribution of Cl between species at 96 moles of oxygen per 100 moles of chlorinator waste. The fraction of Cl associated with the species is graphed on the primary Y axis and the fraction of Cl<sub>2</sub> gas recovery on the secondary X axis.

Fig 4 is a graph of the fraction of Cl present as certain species verses the input of oxygen gas in a thermodynamic simulation of the effect of O<sub>2</sub> on the distribution of Cl between species at 650°C.

Fig 5 is a schematic diagram of a dechlorination facility capable of use in the present invention.

Fig 6 is a schematic diagram of the overall process.

By way of explanation of the invention we illustrate in Figure 2 typical back scatter electron images of (a) a synthetic rutile particle and (b) a carbon particle typical of those present in a sample of blown over material from an operating chlorinator. The images show that the condensed chlorides were present as a coating, about 10µm thick on the surface and in the external pores of the particles. Both images also show small, separate agglomerates 1 to 10 µm of chlorides. Based on examination of the sample under the SEM and microprobe, it appears that about half of the condensed chlorides was present on the particles and the other as agglomerates. It is clear from the nature of the coating on the surface of the rutile and coke particles that the particles cannot be easily



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separated from the chlorides and returned to the chlorinator.

It is important to note that typical samples contained a large number of elements in significant quantities, as chlorides and oxides. Typical concentrations of various species are given in Table 1. For this waste, the stoichiometric oxygen required to completely convert the chlorides and carbon to obtain chlorine and carbon dioxide respectively is about 76 moles of oxygen per 100 moles of waste.

It is noteworthy that the composition of typical waste from a chlorinator is quite different from the simple iron chloride material considered in the prior art. It is also noteworthy that typical waste does not contain any significant quantity of ferric chloride. The major chloride bearing species is ferrous chloride. The large number of chloride and oxide species is particularly noteworthy. The sample also contains a significant amount of carbon, about 23% carbon in the example in Table 1. Prior art does not reveal the conditions required to maximize chlorine recovery and minimize carbon combustion from this type of complex material to obtain chlorine gas with practically relevant concentrations.

By way of the basis for our process we illustrate in Figure 3 the thermodynamic simulation of the results of dechlorination showing the effects of temperature at 96 moles of oxygen per 100 moles of waste. Increasing the temperature decreases the recovery of chlorine due to the increased stability of the chlorides at the higher temperature. At temperatures below 150°C, the only chlorides that do not release chlorine are those of Ba, Na and Nb. At 150°C oxides of vanadium and chromium form. As the temperature increases, the oxychlorides of these metals form, and chlorine recovery is complete from the vanadium and chromium compounds at 400 and 700°C respectively. At 650°C,  $\text{MnCl}_2$  forms a liquid at the expense of  $\text{Mn}_2\text{O}_3$ . As the temperature increases to 1000°C, ferric chloride, and

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manganese chloride increase at the expense of oxides. From 100 to 1000°C, chlorine is completely removed from chlorides of Al, Mg and Ti and not removed from chlorides of Na and Nb.

5

Table 1. Composition of a chlorinator waste

Species	Concentration (wt%)
FeCl <sub>2</sub>	12.4
AlOCl	3.8
MnCl <sub>2</sub>	3.9
MgCl <sub>2</sub>	2.2
TiCl <sub>4</sub>	0.35
V, Cr, Ca, Nb, Zr, Na, Ba, Ce, Sr, Si, Be, Cu chlorides	1.6
TiO <sub>2</sub>	46.1
SiO <sub>2</sub>	2.1
Oxides of Nb, Al, Cr, V, Zr, Cu, Fe, Ce, Ba, Mg, Mn, Ca, Be, Sr, Na	3.2
Carbon	23.2
Sulphur	1.1

10

With increase in temperature the overall equilibrium conversion to chlorine gas decreases but the rates of the various dechlorination reactions increase. These results and our observations show that at higher temperatures where the dechlorination kinetics of ferric chloride are acceptable, a proportion of the iron chloride and manganese chloride remain unreacted and the overall chlorine recovery drops significantly with increase in temperature above about 750°C.

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Figure 4 shows that at a constant temperature of 650°C, increasing the quantity of oxygen increases the recovery of chlorine when thermodynamic equilibrium prevails. Below about 70 moles of oxygen per 100 moles of waste, most of the chlorides remain unreacted due to the preferential consumption of oxygen for the oxidation of carbon. It is clear that above about 85 moles of oxygen per 100 moles of waste, there is no significant increase in the recovery of chlorine.

Results typically as illustrated above clearly show that it is necessary to maximize the recovery of chlorine while minimising the combustion of carbon in order to obtain a chlorine-rich gas and minimize the oxygen consumption.

We have discovered that the carbon combustion can be significantly reduced by the appropriate selection of temperature, gas superficial velocity in the dechlorination reactor and the stoichiometric ratio, R.

A dechlorination facility as illustrated schematically in Figure 5 was used to demonstrate our process. It should be noted that the specific scheme shown in the figure is only indicative of the process used to demonstrate our invention and is not intended to be limiting of the invention in any way, the major purpose of the experimental scheme being the demonstration of the selectivity which was achieved with respect to maximizing chlorine recovery and minimising carbon combustion.

The system used to demonstrate the process comprises a feed hopper 11, an inlet 12 for chlorinator waste and a vent 13. The feed hopper 11 has an outlet 14 which includes an inlet 15 for dry nitrogen and an inlet 16 for oxygen, and hence a mixture of chlorinator waste and a gas containing oxygen is fed along line 17 to the dechlorinator 20. The chlorinator waste 17 is fed into the bottom of the fluidised bed reactor 20 through a conical entry 21 which allows the waste to be injected into the bed in a controlled and reproducible manner. In the

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experiments conducted the bed material was initially rutile which was gradually replaced by the waste material as the test progressed. The bed height was maintained at a constant value by periodically withdrawing accumulated material from the reactor 20. The reactor 20 includes provision to extract or supply heat, in this case cooling the inlet 22 and water cooled jacket 23, as desired to maintain the temperature of the reactor and the fluidised bed at the selected value. Appropriate temperature measurement devices 24 and pressure measurement devices 25 are included.

The fluidised bed reactor 20 includes an outlet 26 for a chlorine-rich gas stream leaving the reactor. The chlorine-rich gas stream is then treated to remove any contaminants from the chlorine gas, first by being passed through cyclone 30 which separates particulate matter from the gas stream, then through filter 31 which removes further small particles from which gas stream 32 emerges which contains chlorine, carbon dioxide and oxygen. This gas stream could be treated if required to increase the chlorine concentration further. In some tests a quencher (not shown) was used before the filter to quench any unreacted chloride vapors.

With this experimental system we achieved independent and careful control of the temperature of the reaction, the oxygen:waste feed ratio, gas superficial velocity and inlet gas composition in the dechlorinator. Other variations were made in the facility when required for specific tests.

By way of examples of the results that demonstrate our invention, the results of various tests are given below. In each of these tests the waste material containing the chlorides typically as listed in Table 1 was fed into the dechlorinator with oxygen under controlled, steady conditions. The products were analysed and the percent chlorine recovery and carbon conversion ( $=100 \times$  chlorine or carbon transferred to the gas phase/chlorine or

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carbon in the feed) were determined. The distribution of residual carbon and chlorine among the condensed products and gas were also determined. Typical results which illustrate the effects of various parameters are given in  
5 Tables 2-4.

In tests 1 and 2, gas at a superficial velocity of 0.5 m/s, calculated from the flow of oxygen in the reactor at the reaction temperature, and a constant oxygen: feed stoichiometric ratio, R, of approximately 0.78 was  
10 used and the effect of varying the temperature is shown in Table 2. We show that the carbon combustion dropped significantly from 58% to 16% when the temperature was reduced from 615 to 600°C thereby producing a gas much  
15 richer in chlorine. The results also show that the chlorine recovery increased significantly with temperature with a recovery of 85% obtained at 615°C.

In tests 3 and 4 the effect of increasing the superficial velocity was investigated at constant temperature and stoichiometric ratio (R = 1 plus or minus  
20 0.07). Again we demonstrate in Table 3 that by increasing the superficial velocity the carbon conversion was decreased to as low as 14% thereby allowing a richer chlorine gas to be produced.

25 Table 2. Effect of temperature

	Test 1	Test 2
Temperature (°C)	615	600
Gas superficial velocity (m/s)	0.5	0.5
Stoichiometric ratio	0.80	0.76
Chlorine recovery (%)	85	62
Carbon conversion (%)	58	16

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Table 3. Effect of gas superficial velocity

	Test 3	Test 4
Temperature (°C)	600	600
Superficial velocity (m/s)	0.7	0.3
Stoichiometric ratio	1.07	0.930
Chlorine recovery (%)	69	85
Carbon conversion (%)	14	33

As a further illustration of the invention, the results of tests 5 and 4 are compared in Table 4. In this case a constant temperature and superficial velocity were maintained and increasing the stoichiometric ratio increased the chlorine recovery to 85% and reduced the carbon conversion from 36 to 33%, again leading to the production of a richer chlorine gas.

Table 4. Effect of stoichiometric ratio, R.

	Test 5	Test 4
Temperature (°C)	600	600
Superficial velocity (m/s)	0.3	0.3
Stoichiometric ratio, R	0.53	0.93
Chlorine recovery (%)	55	85
Carbon conversion (%)	36	33

It should be emphasised that the results in Tables 2-4 are only quoted by way of example to illustrate that the invention gives us the ability to independently control the carbon combustion and chlorine conversion to produce chlorine gas of the desired concentration depending on the application and usage of chlorine.

We have also demonstrated that the invention provides a means for stripping the chloride coating from the carbon particles during their residence in the fluidised bed and that the unreacted carbon blown over and collected in the cyclone is substantially free of chloride.

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The chlorine from the chloride coating and separate chloride particles react with the oxygen in the gas and chlorine is efficiently separated from the carbon particles.

5           By way of example, in Test 2, a low carbon conversion of 16% was obtained. Of the unconverted carbon, 88% was collected in the cyclone thereby achieving an efficient separation of the carbon and only a small loss of carbon as carbon dioxide into the gas phase. In Tests 1  
10       and 5, 95% and 89% respectively of the unreacted carbon was collected in the cyclone and confirm that the application of the invention results in good collection efficiencies of the unreacted carbon in the cyclone. These examples are  
15       quoted to illustrate the invention and are not intended to be limiting in any way.

          A further aspect of our invention is that it also provides a means for increasing the chlorine recovery from the waste by separating the blown over carbon and rutile particles in the cyclone at a temperature above the  
20       condensation temperature of any unreacted chloride which avoids the condensation of these chlorides in the cyclone. The temperature of the cyclone will depend on the type of chloride vapors in the exit of the dechlorinator. By  
25       maintaining the temperature of the cyclone above the condensation temperature of the vapors, the unreacted vapors are separated from the carbon and rutile particles. For example a temperature of about 350°C ensures that ferric chloride vapour does not condense in the cyclone.

          A further aspect of the invention is that the  
30       upper limit of the temperature of the cyclone is determined by the need to prevent the oxidation of carbon in the cyclone. This will depend on several factors including the carbon content of the material collected in the cyclone, the oxygen potential of the gas, determined by its CO<sub>2</sub> and  
35       oxygen concentration, the reactivity of the carbon in the gas and the residence time of particles and gas in the cyclone. In typical operation with a waste of the

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composition and characteristics in Table 1 we have found that cyclone temperatures from 400 to 600°C are satisfactory and preferably from 450 to 550°C to ensure negligible carbon oxidation. However, it should be emphasised that the present invention is not limited to these temperatures due to the related considerations mentioned above.

We have also found that the unreacted chloride vapors can be condensed in an appropriately designed condenser to produce a condensed chloride product substantially free of carbon and rutile. The design conditions for the condenser can be easily determined from the knowledge of the hot gas and vapour compositions and flow rates arriving at the quencher and the temperature which determine the amount of heat to be extracted to cool the gas and the surface area required to achieve efficient condensation.

As a further aspect of our invention it is possible to treat this condensed product which is substantially chloride either by recirculating it to the fluidised bed reactor or by treating it with oxygen in a separate reactor to increase the recovery of chlorine.

Our invention therefore relates to the novel processing steps which maximize the recovery of chlorine, minimize the conversion of carbon, and separate the unconverted carbon and rutile and other inert materials from the waste in an appropriately designed cyclone.

The overall process is illustrated in the flow chart given in Fig 6. The process comprises feeding chlorinator waste with a gas containing oxygen to the fluidised bed dechlorinator 20 in which the conditions of temperature, gas velocity and oxygen to feed stoichiometry are controlled in an appropriate manner to optimise the recovery of chlorine and minimize the conversion of carbon. This dechlorination step essentially separates the chlorine from the chloride coating associated with carbon and rutile particles contained in the chlorinator waste. A chlorine



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rich vapour stream leaves the dechlorinator and is passed to a cyclone 30 in which any particles of carbon and rutile which leave the dechlorination reactor (having been stripped of chlorine in the reactor) are separated from the gas stream. The cyclone is maintained at a temperature above the condensation temperature of any unreacted chlorides so that an effective separation is achieved between the solids and the unreacted chloride vapors. Any unreacted chloride vapors remain in the chlorine-rich gas stream and are conveyed to the quencher 40. In the quencher any unreacted chlorides are condensed but the chlorine gas stream passes through the quencher to a baghouse or other appropriate separation device 31 to remove any residual fine dust particles so as to obtain a clean gas. The quencher condenses chlorides, which are substantially free of carbon, rutile and other solids and may be recirculated to the dechlorination reactor or may be reacted separately with oxygen to recover the residual chlorine. Thus, the process offers a practical means of increasing the chlorine recovery.

Throughout this specification and the claims, the words "comprise", "comprises" and "comprising" are used in a non-exclusive sense.

Modifications to the process for chlorine recovery described above apparent to the person skilled in the art and within the spirit and scope of the present invention are encompassed by the above description.

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Claims:

1. A process for recovering chlorine from chlorinator waste including the steps of:
  - 5 forming a fluidised bed of chlorinator waste in a fluidising gas containing oxygen and treating the chlorinator waste with oxygen under conditions which promote conversion of metal chlorides into metal oxides and discourage oxidation of carbon contained in the waste.
- 10 2. A process according to claim 1, wherein the conditions which promote conversion of metal chlorides into metal oxides and discourage oxidation of carbon contained in the waste are obtained by controlling the superficial velocity of the fluidising gas, the proportion of oxygen in the gas fed to the fluidised bed, oxygen to chlorinator waste feed ratio and temperature within the fluidised bed either separately or in combination.
- 15 3. A process according to claim 2, wherein the temperature of the fluidised bed is maintained in a range from 400 to 700°C.
- 20 4. A process according to claim 2 or claim 3, wherein the superficial velocity of the gas is maintained in a range from 0.2 to 1 metre/second.
- 25 5. A process according to any one of claims 2 to 4, wherein the stoichiometric ratio, R, is maintained in a range from 0.2 to 1.2.
6. A process according to any one of claims 2 to 5, wherein the temperature of the fluidised bed is maintained in a range from 550 to 650°C.
- 30 7. Apparatus for recovering chlorine from chlorinator waste, which apparatus includes a fluidised bed reactor, means for introducing chlorinator waste into the fluidised bed reactor, means for introducing a fluidising gas containing oxygen into the fluidised bed reactor and means for controlling oxygen to chlorinator waste molar feed ratio, superficial velocity of fluidising gas, proportion of oxygen in the fluidising gas and temperature
- 35

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within the fluidised bed reactor.

8. Apparatus according to claim 7, including means for collecting particulate matter which leaves the fluidised bed reactor and means for quenching any unreacted chloride compounds.

9. Apparatus according to claim 8, wherein the means for collecting particulate material is a cyclone.

10. Apparatus according to claim 9, wherein the apparatus includes means for maintaining temperature in the cyclone above a level at which metal chlorides would condense but below that at which significant quantities of carbon would oxidise.

11. Apparatus according to claim 10, wherein the temperature of the cyclone is maintained in a range from 400 to 600°C.

12. A process for recovering chlorine from a mixture containing a metal chloride and carbon, the process including forming a fluidised bed of the mixture in a fluidising gas containing oxygen and converting the mixture under conditions which promote conversion of metal chlorides to metal oxides and discourage oxidation of carbon.

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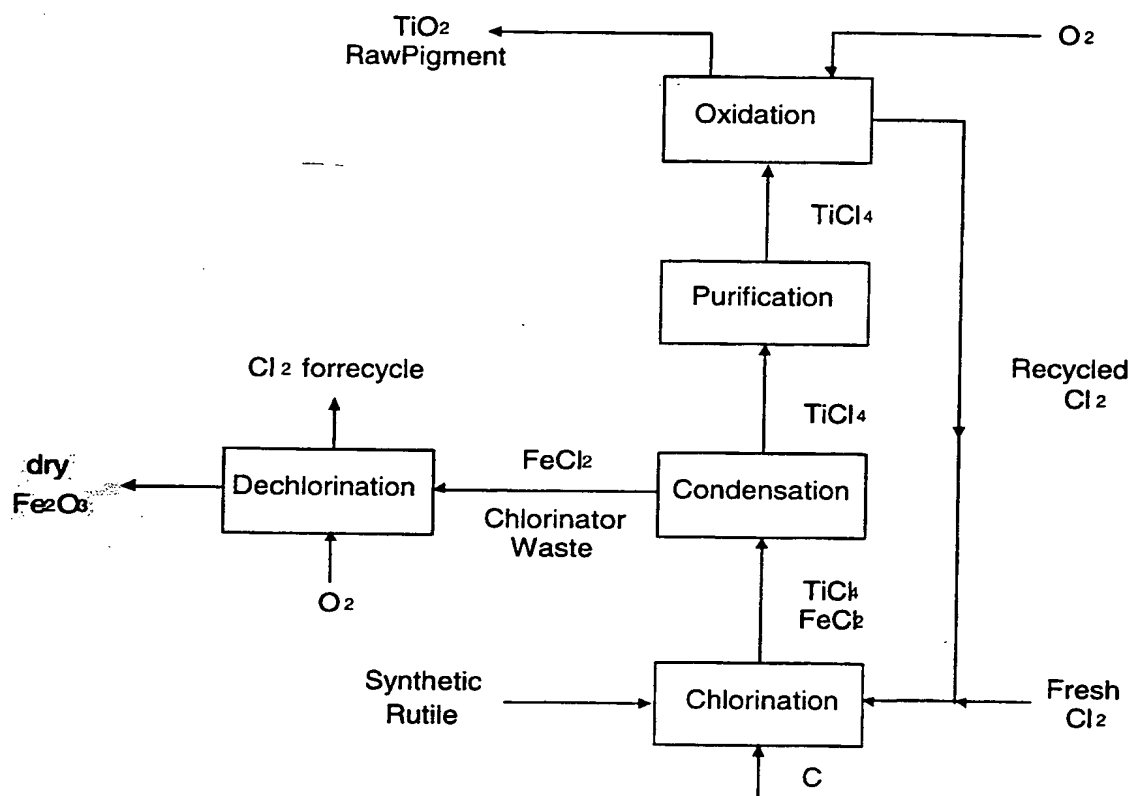


Figure 1

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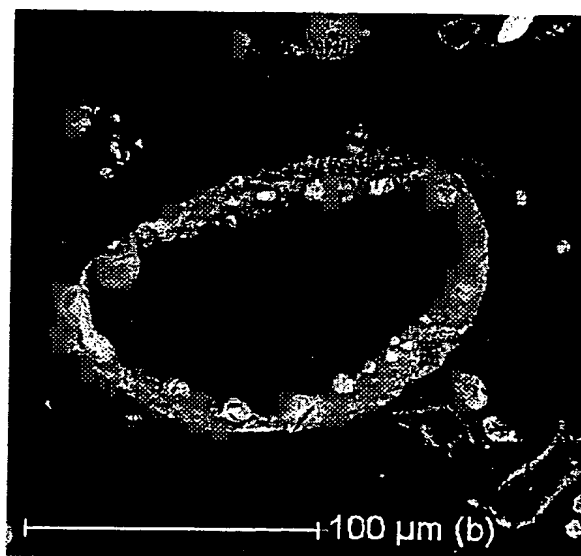
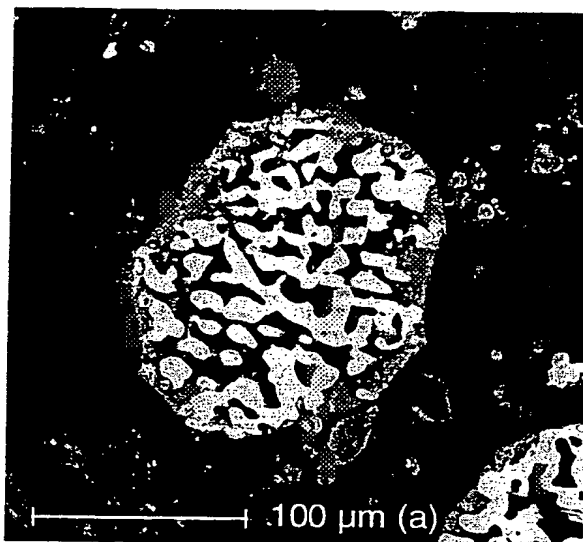


Figure 2

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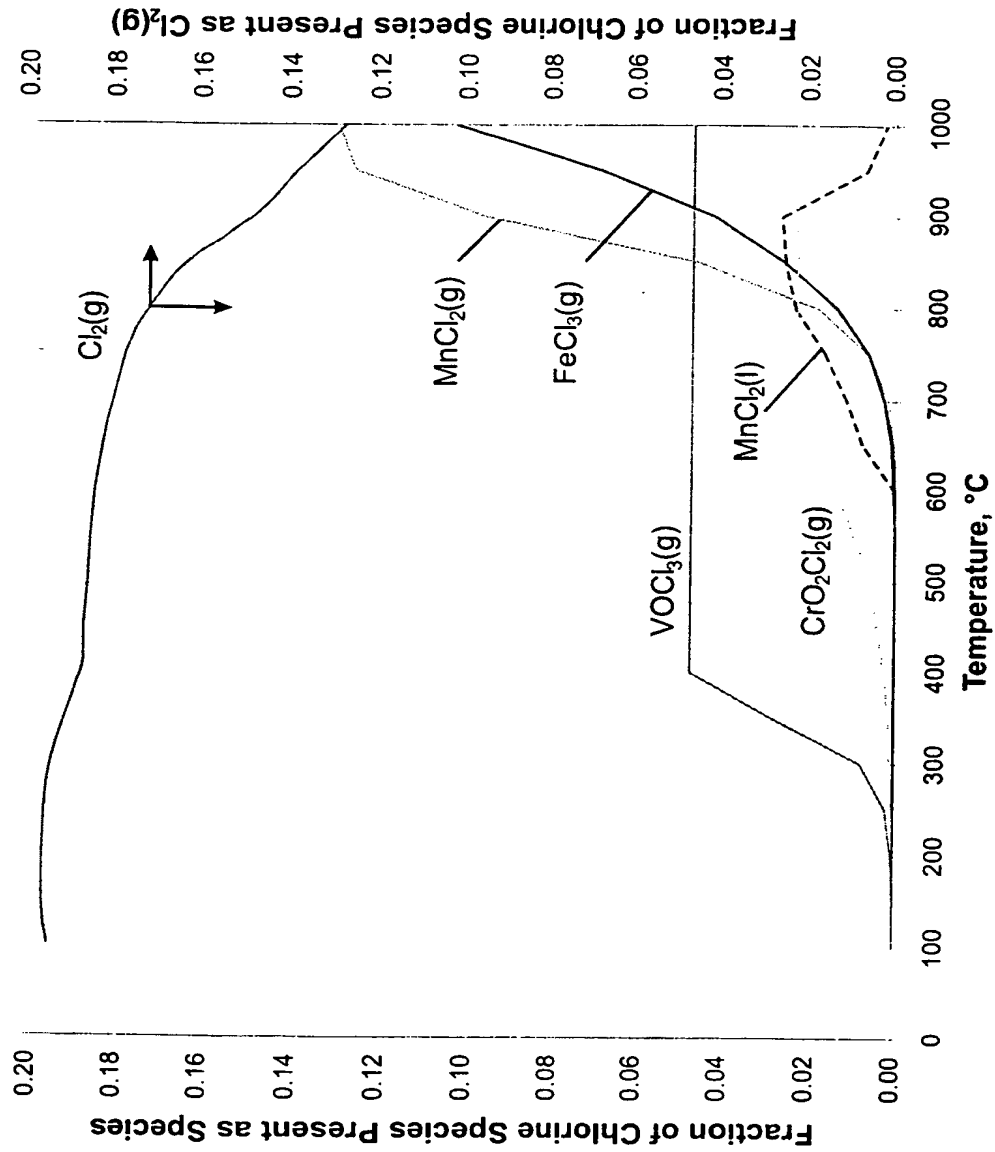


Figure 3

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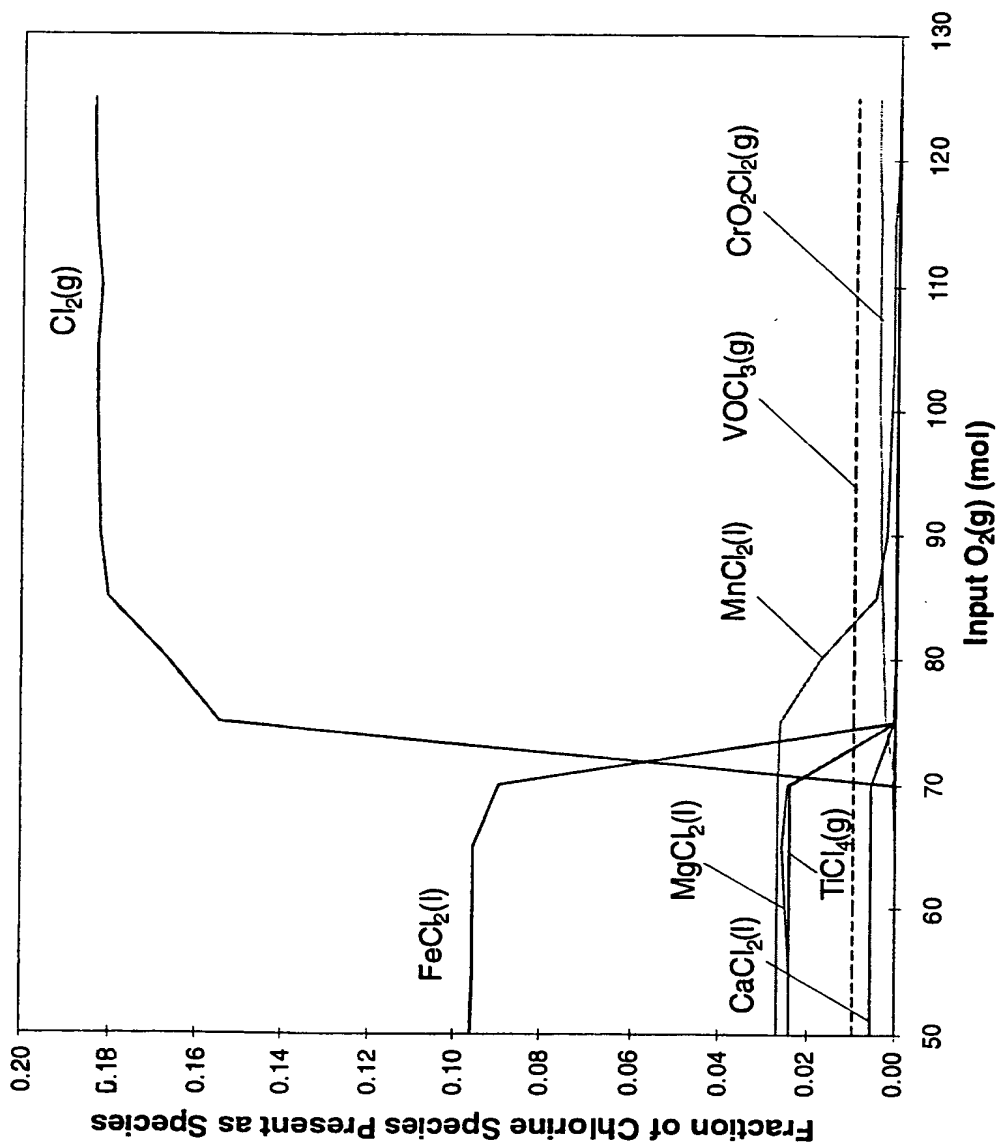


Figure 4

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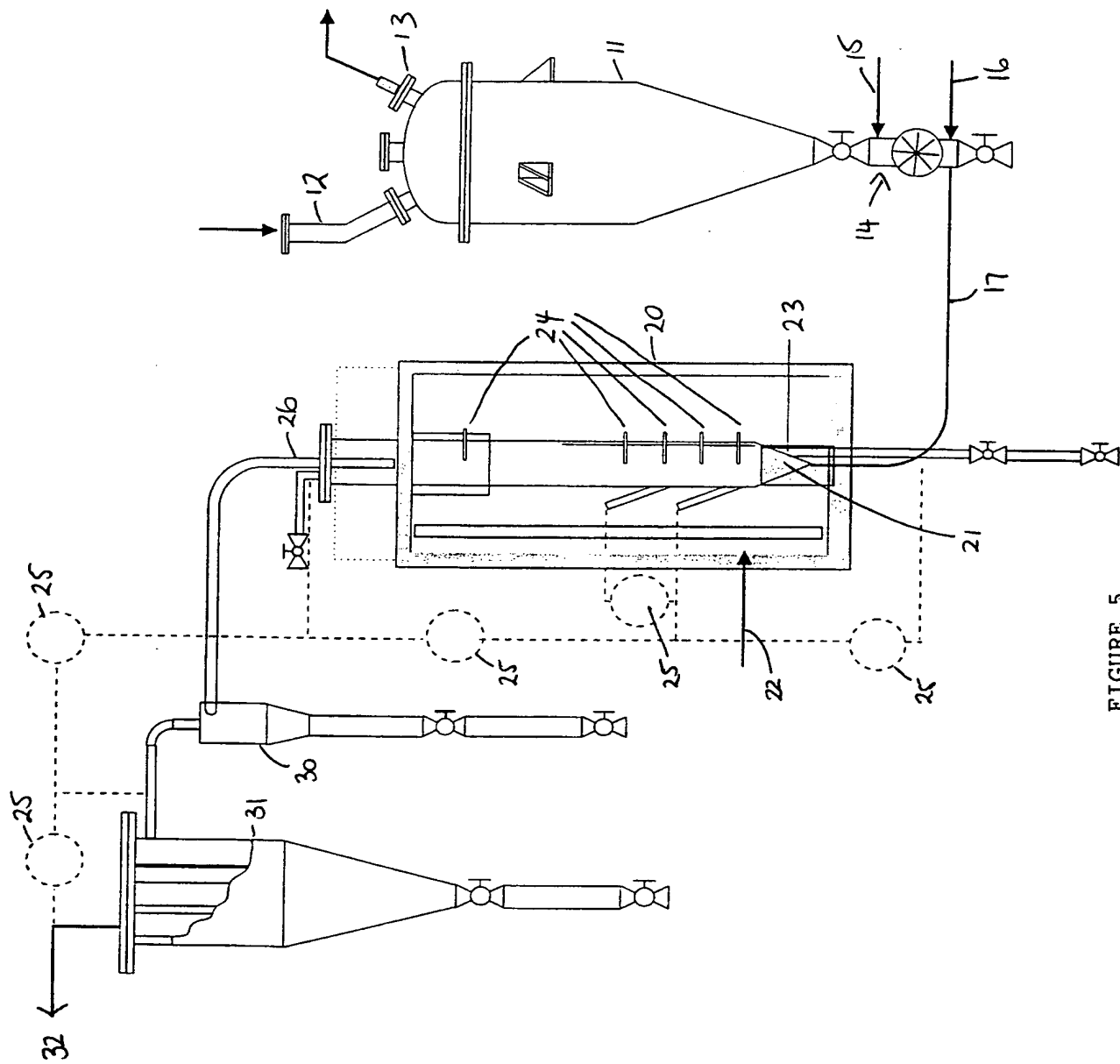


FIGURE 5



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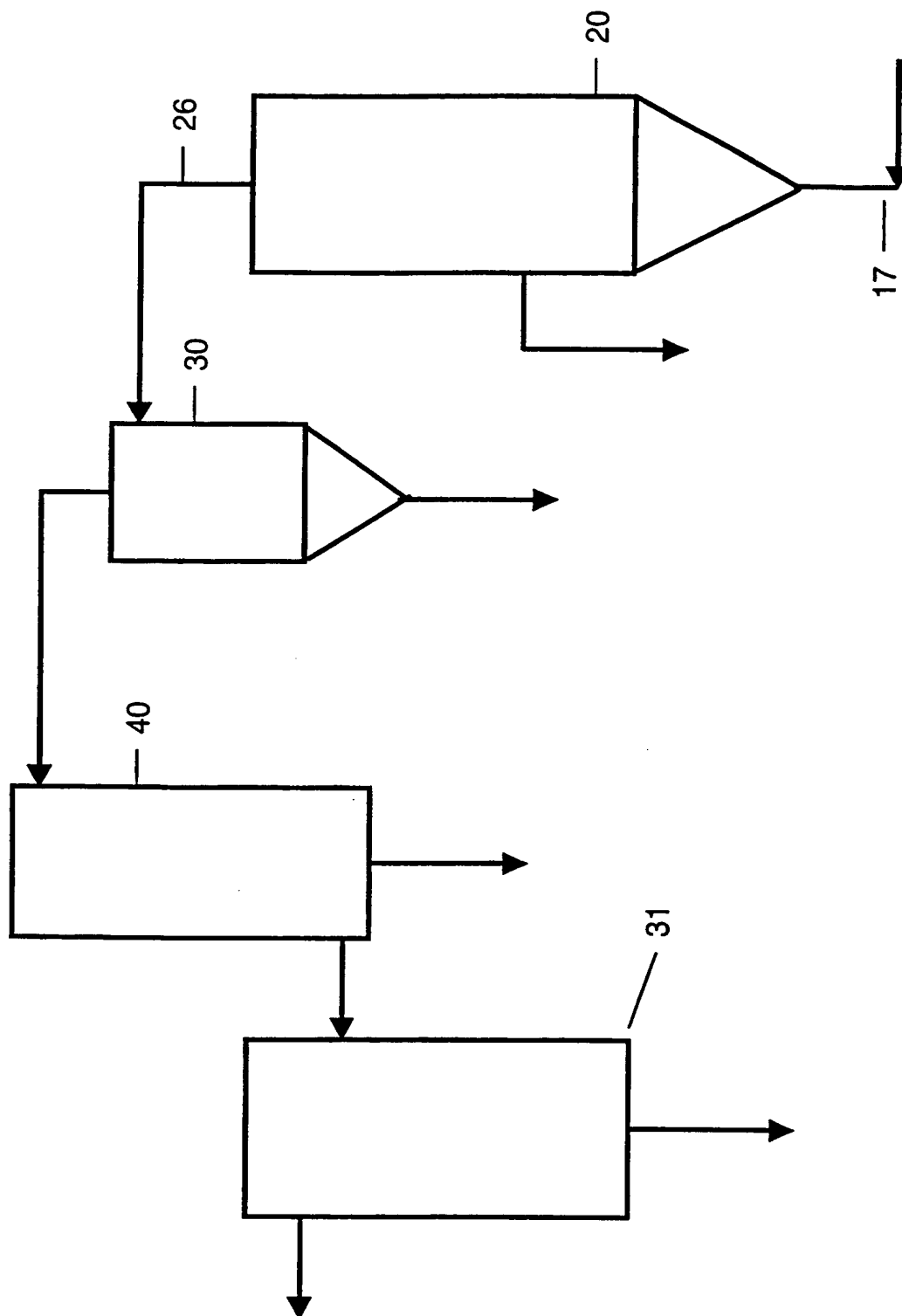


Figure 6

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/AU 99/00546

## A. CLASSIFICATION OF SUBJECT MATTER

Int Cl<sup>6</sup>: C01B 7/03, C01G 49/06, C22B 34/12

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
C01B 7/03, 7/02 : C01G 49/06, 49/02, 49/04 ; C22B 34/12

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
AU:IPC as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
WPAT: IRON:OR FER: CHLORI:REGEN: OR RECOVER: OR RECYCL: OF REUS: OR WASTE:

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	AU 30628/77 A (TURNER JHW AND SHACKLETON C.E.E) 24 May 1979 Pages 2 lines 1-4 page 5 line 15 page 6 line 13	1-6, 7-12
X	AU 46786/79 A (SHACKLETON C.E.E) 15 November 1979 page 2 line 1-4 page 6 line 15 page 7 line 15.	1-6, 7-12
X	US 4994255 A (CHARLES K. HSU) 19 February 1991 Column 1 line 12 to 14 column 4 line 45 to 68	1-6, 7-12

☒ Further documents are listed in the  
continuation of Box C

☒ See patent family annex

<p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier application or patent but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p>		<p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&amp;" document member of the same patent family</p>
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Date of the actual completion of the international search  
29 July 1999

Date of mailing of the international search report  
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Name and mailing address of the ISA/AU  
AUSTRALIAN PATENT OFFICE  
PO BOX 200  
WODEN ACT 2606  
AUSTRALIA  
Facsimile No.: (02) 6285 3929

Authorized officer  
  
**ROGER HOWE**  
Telephone No.: (02) 6283 2159

**INTERNATIONAL SEARCH REPORT**  
**Information on patent family members**

International application No.  
**PCT/AU 99/00546**

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report				Patent Family Member			
AU	30628/77	CA	1085589	DE	2751434	IT	1092208
		JP	53063299	US	4140746	CA	1119384
		FR	2425403	GB	2020643	JP	55003381
		US	4259298	DE	2918945		
AU	46786/79	CA	1119384	DE	2918945	FR	2425403
		GB	2020643	JP	55003381	US	4259298
							END OF ANNEX